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# EFFECT OF SIZE ON TENSILE STRENGTH OF FINE POLYCRYSTALLINE NICKEL WIRES

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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## ABSTRACT

Small-diameter (250 to 10  $\mu\text{m}$ ) polycrystalline nickel wires were prepared from single crystals and tested to relate tensile properties to wire size. Four conditions were tested: cold-drawn; drawn and electropolished; drawn and recrystallized; and drawn, recrystallized, and electropolished. The drawn wires were tested as a function of wire diameter, and recrystallized wires were tested at two grain sizes. The 0.2-percent yield strengths and tensile strengths of cold-drawn wires varied inversely as the square root of wire diameter. The tensile strength of recrystallized wires was diameter independent. The proportional limit stress of recrystallized wires was inversely related to power functions of wire diameter and grain size. This relation can be expressed by a modified Hall-Petch equation.

# EFFECT OF SIZE ON TENSILE STRENGTH OF FINE POLYCRYSTALLINE NICKEL WIRES

by Lester S. Rubenstein  
Lewis Research Center

## SUMMARY

Polycrystalline nickel wires with diameters from 254 microns to less than 10 microns were prepared from single crystals by various combinations of drawing and electropolishing. The wires were tested in four conditions: cold-drawn; drawn and electropolished; drawn and recrystallized; and drawn, recrystallized, and electropolished. All wires were tested as a function of wire diameter, and the recrystallized wires were tested at two grain sizes.

The 0.2-percent yield strengths and tensile strengths, obtained for drawn wires by a sequential testing technique, varied inversely as the square root of wire diameter. The ultimate tensile strengths obtained for recrystallized wires were independent of wire diameter. The proportional limit stress of recrystallized wires was inversely related to power functions of wire diameter and grain size. This stress relation is indicated by an increasing slope of Hall-Petch plots with decreasing diameter. This increase has been interpreted as reflecting a decrease in average spacing of dislocation barriers with both decreasing wire diameter and decreasing grain diameter. The relation can be expressed by the following modified Hall-Petch equation:

$$J_{PL} = J_0 + K_y D^{-1} L^{-1/2}$$

where  $J_{PL}$  is the proportional limit stress,  $J_0$  is the friction stress or zero intercept of the Hall-Petch plot,  $K_y$  is the slope of the Hall-Petch plot,  $D$  is the wire diameter, and  $L$  is the grain size.

## INTRODUCTION

The tensile strengths of metal whiskers (ref. 1), vapor-deposited thin films (ref. 2), foils thinned from the bulk material (ref. 3), and small diameter monocrystals (ref. 4) have been shown to depend inversely on diameter or thickness (ref. 5). For example, Brenner showed that some whiskers that were less than 4 microns in diameter exhibited resolved elastic shear strengths of from 2 to 6 percent of their shear moduli (ref. 1) and that they exhibited strengths related to whisker size. The explanations for size dependence of whiskers have varied. Some that have been suggested are the absence of mobile dislocations in small diameter metal whiskers (refs. 1 and 5), the formation of excess point defect concentrations during vapor deposition of thin films (ref. 2), the effect of image forces or surface films on dislocation egress or motion in single crystal or foil specimens (refs. 6 to 8), and finally changes in the effective length of the glide plane which contains the burgers vector of the mobile dislocations (refs. 9 and 10).

An important consideration that adds impetus to studies on the size dependence of fiber strength is that their high strength makes them desirable as reinforcements in composite materials (ref. 11). Polycrystalline wires are of special interest for composite material applications as they are produced with relative ease, with high yield and tensile strengths. In addition, wires do not easily lose their strength in handling, have controllable length to diameter ratios, and are easily incorporated into a composite matrix.

The purpose of this investigation was to study the effects of wire diameter, grain size, cold work, and electropolishing on the room-temperature tensile properties of polycrystalline nickel wires in the diameter range of 1 to 200 microns.

## MATERIALS, APPARATUS, AND PROCEDURES

### Wire Purity and Texture

High-purity single-pass zone-refined nickel with impurity content as listed in table I was used. The wires were obtained by drawing from three single crystals of different orientations, originally 0.125 inch (0.317 cm) in diameter, in an attempt to control the texture (ref. 12). One crystal was oriented with its long axis within  $3^\circ$  of the [111] corner of the standard stereographic triangle, a second within  $3^\circ$  of the [100] corner, and the third halfway between the two corners and adjacent to the line joining the two corners. The wires were cold-drawn to 254, 127, 76.2, 38.1, 20.3, and 15.2 microns. These were drawn without stress relief or other anneal treatment. Samples were obtained at various steps during the drawing operation to yield a selection of wire from each of the drawing stages. The true drawing strains (natural log of the ratio of initial to final cross

TABLE I. - CHEMICAL ANALYSIS OF ZONE

## REFINED NICKEL WIRES

Impurity element	Impurity content, ppm
Silicon	0.001
Sulfur	.001
Manganese	.001
Iron	.001
Nitrogen	.03
Carbon, total	.03

section area) of the wire ranged from 5.5 to 11.0.

Careful comparison of the intensities of the Debye rings on the film indicated that there were no texture differences in the wires drawn from the variously oriented crystals. The X-ray photographic method used was representative of an integrated area of the wire surface after electropolishing to remove at least 15 microns from the surface diameter. Good symmetry was visually observed around the Debye rings of the film.

Examination of the surfaces of the cold-drawn wires showed that they were composed of a fibrous structure with the fibers aligned parallel to the drawing direction. A typical electron micrograph representative of the electropolished and etched surface of the 127 micron diameter cold-drawn wire is observed in figure 1.

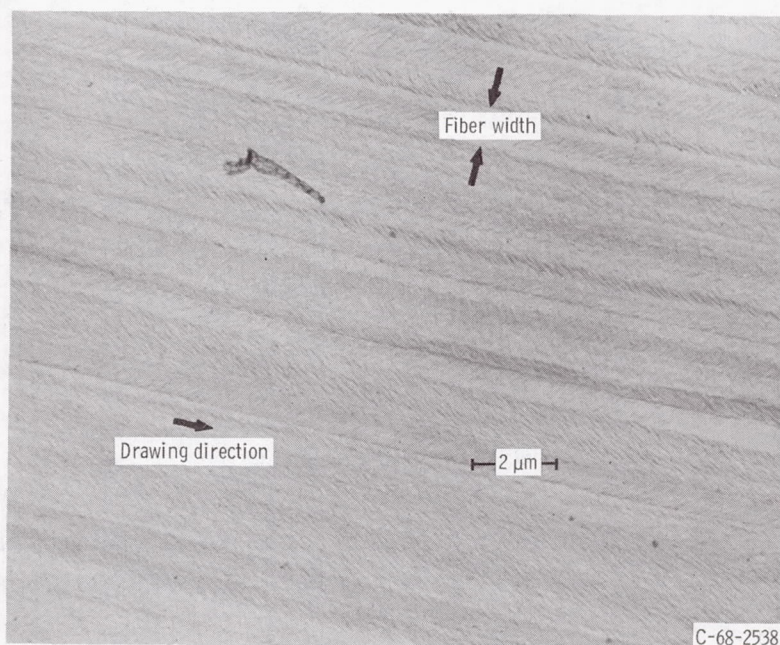


Figure 1. - Electron micrograph of drawn wire.



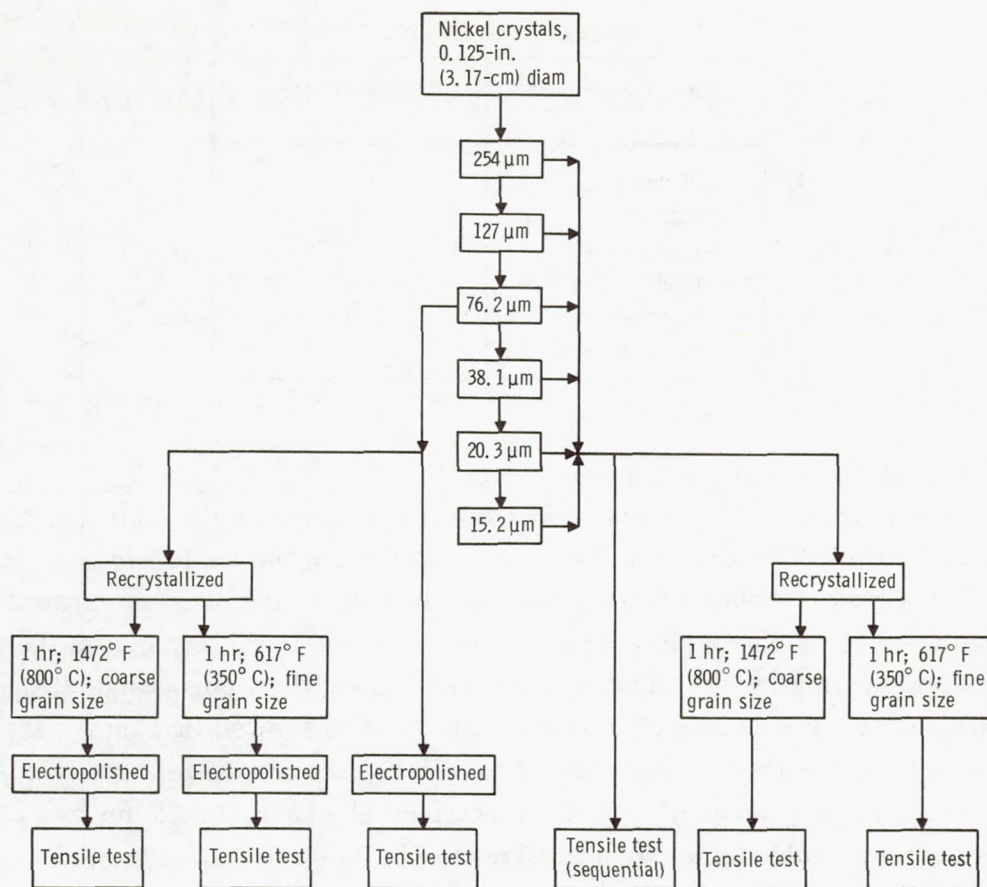


Figure 2. - Processing of nickel wire.

## Wire Conditions Evaluated

The drawn wires were tested in four major conditions as shown in the flow chart in figure 2. The four conditions were (1) cold-drawn, (2) cold-drawn and electropolished, (3) cold-drawn and recrystallized, and (4) cold-drawn, recrystallized, and electropolished.

## Diameter Measurement

Wire diameters were measured and checked for cross-sectional uniformity with an image-splitting measuring eyepiece. The wires were measured with a magnification power of 300X.

Cross-sectional uniformity was also determined using the image splitting eyepiece.

When the double images were exactly edge to edge in the axis of desired measurement, the microscope stage was translated perpendicular to the sighting direction to scan the wire along its diameter. Those sections of wire larger than the section initially measured appear with double images overlapping (black); sections of wire with smaller diameters appear with double images apart. By thus scanning the gage length, the minimum diameter can be located and cross-sectional uniformity determined.

## Electropolishing Technique

The electrolyte was a phosphoric acid - sulfuric acid solution. Using this solution it was possible to obtain optically smooth surfaces at 300 magnifications for wires whose diameters were relatively uniform. Smooth surfaces and uniform diameters were achieved by oscillating the specimen through  $25^{\circ}$  of arc in a magnetically stirred electrolyte maintained at  $125^{\circ}$  F ( $52^{\circ}$  C). To control specimen thickness, metal removal was monitored with a stop watch using curves of material removed against time for various initial diameters of wire. No difficulty was encountered in polishing the recrystallized wires. However, the cold-drawn wires, after an appreciable amount of surface removal, tended to curl during polishing because of the release of surface constraints imposed on the interior residual stresses. This violated the carefully maintained uniform cathode-anode distance and caused preferential polishing. In addition, at sizes less than 10 to 15 microns the "curling tendency" was often strong enough to cause wire failure before the desired amount of uniform metal removal was attained, thus limiting the sizes of the wires available for testing.

## Tensile Testing of Wires

Tensile testing was conducted on a screw driven model test machine at a loading rate of 0.1 inch per minute (0.25 cm/min). Specimen wires were handled with magnetic tweezers and they were mounted in the following manner. The wires were positioned on small metal tabs and fixed in place with bankers sealing wax. This required the handler to wear magnifying lenses to ensure careful manipulation. The tabs were held under spring load to a clear-plastic fixture so that no load could be imposed on the test specimen. The assembly with specimen was then placed in the micromanipulator mounted on the crosshead of the test machine. The gage length of the test machine grips were adjusted so that of the tensile specimen and the tabs were then fixed to the grips. The spring-loaded clips holding the tabs snug to the clear-plastic support fixture were removed and the micromanipulator slowly energized to allow the specimen to hang free.



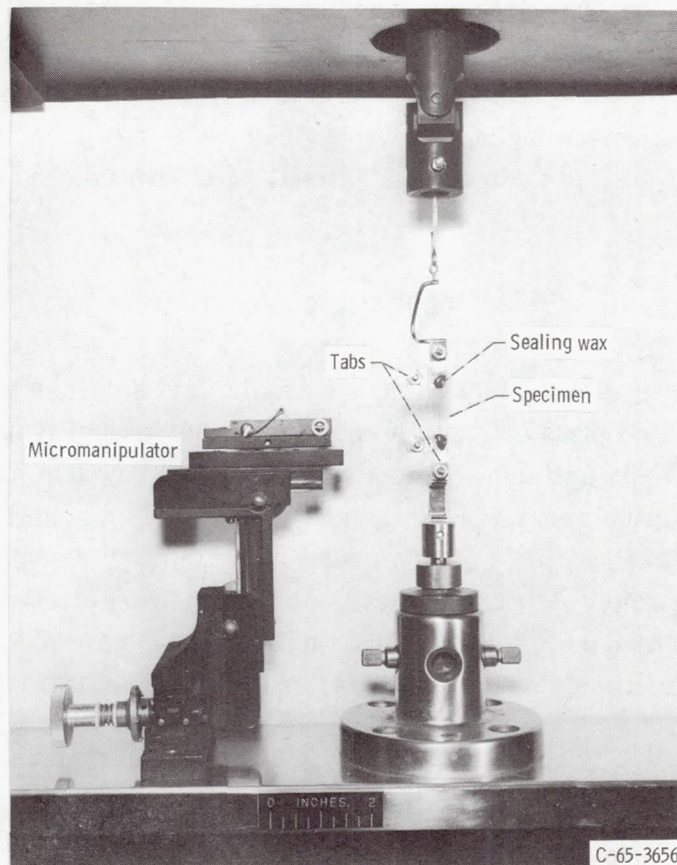


Figure 3. - Specimen test fixtures.

The fixture and specimen may be seen in figure 3 showing its position in the micromanipulator and the testing machines grips. Proportional limit stresses were determined by selecting that point at which the load-elongation curves departed from linearity.

Tensile testing of all groups of specimens was done on 1-inch (2.54-cm) gage length specimens. Such specimens were satisfactory for most of the groups. However, the as-cold-drawn wires displayed a large variation in tensile strength and 0.2 percent yield strengths. In fact, the scatter was so large that these data are not reported herein. Cold-drawn wires were subsequently tested as a function of gage length by a "sequential" testing technique (ref. 13). The starting gage length of the specimen is essentially 6 inches (15.2 cm). After tensile testing the starting specimen, subsequent testing was continued using the longer remaining portion of each successively tested specimen until a gage length too small for further testing resulted. This method allowed the reduction of the effects of surface and internal defects that may have been introduced during handling or cold drawing. The first specimen to fail in a sequence was believed to have been

influenced by flaws and thereby would not represent the true strength of the materials. The sequential testing method also eliminates effects due to sample shapes or grips. Using this method it was possible to get a true elongation of a material by plotting the percent elongation against the reciprocal of the gage length and extrapolating the curve to a reciprocal value of zero.

Tests of recrystallized specimens also were a problem. Some of these materials were embrittled, presumably by the heat treatment or heat-treating atmosphere. Contamination of grain boundaries or other effects could have damaged some of the specimens. Premature failure of the recrystallized materials was eliminated by including only the data for specimens that failed in a ductile manner. Such failures were characterized by a chisel-shaped fracture or an elongated specimen. Cup and cone fractures were considered to be impurity particle nucleated or brittle (refs. 14 and 15), and were not used for the correlations that subsequently will be presented.

All the electropolished wires were thinned to their final diameter for testing from initial cold-drawn or recrystallized diameter of 76.2 microns (see fig. 2). The following procedure was used: The wires were electropolished, rinsed successively in unused electrolyte, deionized water, and ethyl alcohol. They then were examined optically with a stereoscopic microscope for surface flaws. Tensile tests of selected gage lengths were made. The diameters of the specimens were measured with the split-image microscope. As described previously, handling and placing in tensile grips was accomplished using a micromanipulator. The time from start of electropolishing to initiation of tensile testing was kept to less than 20 minutes to provide a reasonably clean test specimen surface and to minimize atmospheric attack.

## Recrystallizing Treatments

Treatments were carried out in a furnace with a tungsten split-filament heating element under a dynamic vacuum of  $1 \times 10^{-7}$  torr, or better. Temperature was controlled to within  $18^{\circ}$  F ( $10^{\circ}$  C). The two annealing treatments, 1 hour at  $617^{\circ}$  F ( $325^{\circ}$  C) and 1 hour at  $1472^{\circ}$  F ( $800^{\circ}$  C) were, designed to form, respectively, a fine grained and coarse grained material. The average fine-grain size was 0.02 millimeter and the coarse-grain size, 0.17 millimeter.

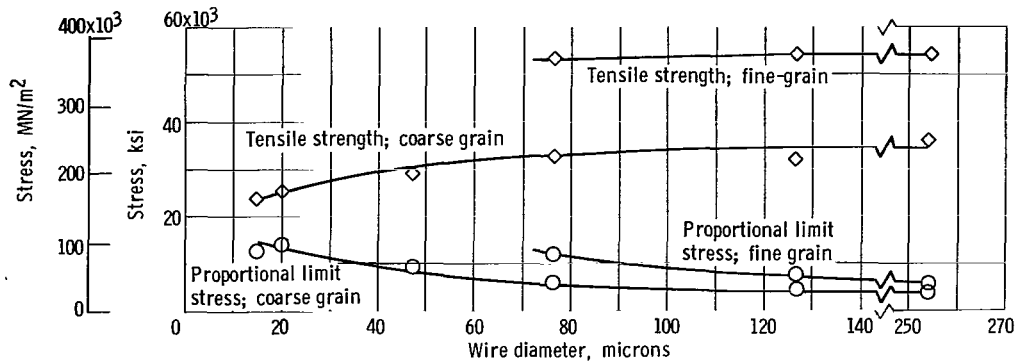


Figure 4. - Effect of wire diameter on proportional limit and tensile strength of recrystallized wire.

## RESULTS

### Recrystallized Wires

A plot of proportional limit and tensile strength against wire diameter for the cold-drawn and recrystallized wire data shows that the proportional limit stress increases and the tensile strength decreases as the wire diameter is reduced (fig. 4). The finer grain material is stronger than the coarser grain material.

Proportional limit stress and tensile strength of the wires that were recrystallized and electropolished from 76.2 microns in diameter to smaller sizes revealed (table II) an inverse dependence of proportional limit stress on size, but the tensile strength is apparently independent of size. Proportional limit stress and yield strength had been correlated with expressions that related these values to wire diameter to the -1 (refs. 1 and 16), -1/2 (ref. 17), or -3/2 (ref. 18) powers. To obtain an exponent for the data of this investigation, a linear regression analysis of a logarithmic plot of proportional limit stress against wire diameter was made. The dependence of this value on the diameters of the fine- and coarse-grained material was determined to be, respectively;

$$\log(J - 4400) = -1.58(\pm 0.19) \log D - 0.65(\pm 0.56) \quad (1)$$

with the correlation coefficient equal to 0.81

$$\log(J - 3000) = -1.22(\pm 0.11) \log D + 0.21(\pm 0.35) \quad (2)$$

with correlation coefficient equal to 0.98 and where  $J$  is the stress and  $D$  is the wire diameter. The values of 4400 and 3000 psi (30.33 and 20.68 MN/m<sup>2</sup>) being, respectively,

TABLE II. - TENSILE PROPERTIES OF RECRYSTALLIZED  
AND ELECTROPOLISHED NICKEL WIRES

Wire diameter, microns	Proportional limit stress		Tensile strength	
	psi	MN/m <sup>2</sup>	psi	MN/m <sup>2</sup>
Coarse-grain material (0.17 mm)				
34.0	7.7	53.1	31.3	215.8
29.7	9.4	64.8	28.0	193.1
25.7	10.0	68.9	31.3	215.8
19.6	14.0	96.5	28.6	197.2
12.9	22.8	157.2	28.4	195.8
9.0	25.7	177.2	40.0	275.8
Fine-grain material (0.02 mm)				
42.9	8.9	61.4	49.1	347.4
42.1	11.6	80.0	39.0	268.9
32.3	10.5	72.4	41.1	283.4
32.0	12.0	82.7	39.8	274.4
30.6	13.0	89.6	36.9	254.4
26.9	17.1	179.0	36.5	251.7
22.0	22.0	168.5	32.3	222.7
21.1	28.1	193.7	44.1	304.1
13.2	44.0	303.4	44.1	304.1
11.6	35.4	244.1	38.1	262.7

the proportional limit stresses for the 254-micron-diameter wires of the fine- and coarse-grained material. This assumes the 254-micron-diameter wire exhibits properties similar to those of bulk nickel.

A regression analysis of the data for the linear forms of these equations gave

$$J = 4400 + 0.226D^{-1.58} \quad (3)$$

and

$$J = 3000 + 1.624D^{-1.22} \quad (4)$$

Figure 5 presents the least squares curves which express the previously noted dependence of proportional limit on diameter for the recrystallized and electropolished wires. The actual data points are included so that a comparison of the calculated- and experimental-strength wire-diameter dependence may be made. The least squares curves were drawn with points obtained by selecting wire diameters which approximated those actually tested and solving the linear regression equations (3) and (4) for the proportional

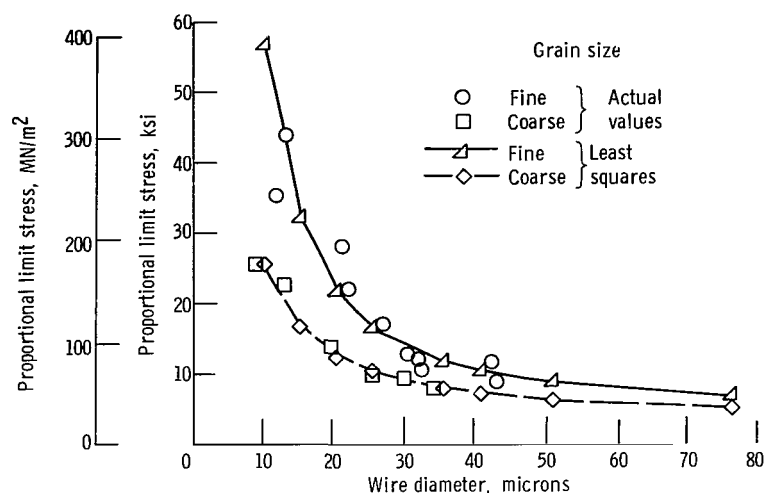


Figure 5. - Comparison of experimental and calculated variation of strength with wire diameter. All wires were recrystallized and electropolished.

TABLE III. - CALCULATION OF PROPORTIONAL LIMIT STRESSES FROM REGRESSION EQUATIONS

Wire diameter, micron	Calculated stress for coarse-grain size material		Calculated stress for fine-grain size material	
	psi	MN/m <sup>2</sup>	psi	MN/m <sup>2</sup>
40.6	7.172	49.45	10.270	79.63
35.6	7.910	54.54	11.650	89.15
25.4	10.400	78.74	16.720	124.11
20.3	12.720	96.53	21.930	160.04
15.2	16.800	124.66	32.000	229.46
10.2	25.630	185.54	56.760	400.17

limit stresses. The appropriate wire diameters and calculated proportional limit stresses may be seen in table III.

The wires that were recrystallized at 76.2-micron-diameter to obtain either coarse or fine grain sizes, all show an increase in proportional limit stresses on electropolishing to smaller diameters. However, as noted previously, the ultimate tensile strength does not vary with wire diameter for either grain-sized material (table II). The tensile strength of the larger grained material averaged  $31.3 \times 10^3$  psi ( $215.8 \text{ MN/m}^2$ ), and the smaller grained material averaged  $40.1 \times 10^3$  psi ( $276.5 \text{ MN/m}^2$ ). The total elongation at fracture of recrystallized and electropolished wire (see table IV) appears to be strongly size dependent, increasing with increasing wire diameter. The maximum

elongation for large grain size wire was 16.1 percent (for 69.7- $\mu\text{m}$  diam), while the maximum for cold-drawn wire was 1.40 percent elongation (for 60.4- $\mu\text{m}$  diam).

## Cold-Drawn Wires

Engineering stress-true elongation plots obtained from sequential testing of the unelectropolished cold-drawn wires are presented in figure 6. Note that total elongation at fracture decreased with increasing prior cold work (decreasing diameter). Ductility

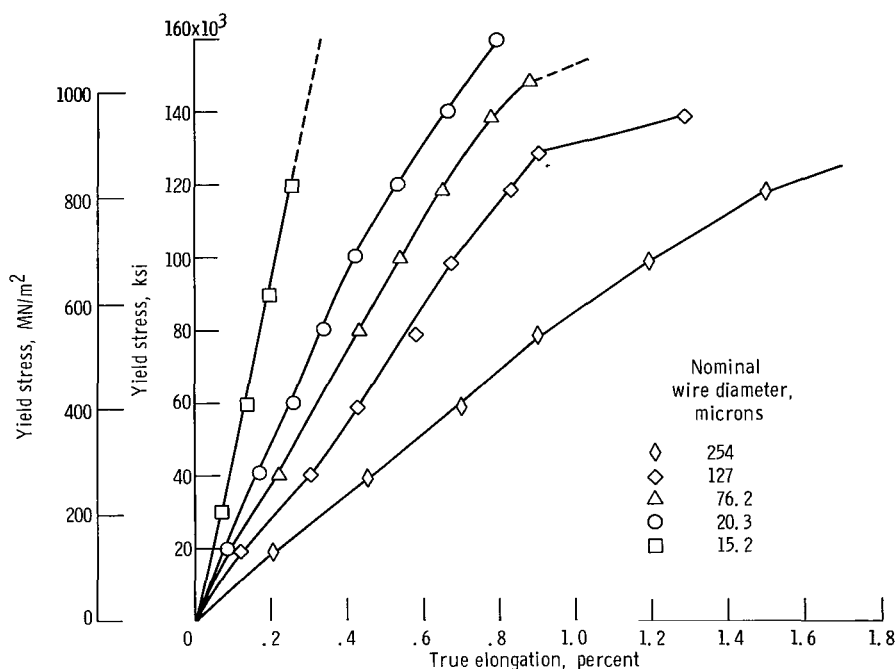


Figure 6. - Yield stress. Elongation data for various diameter wires.

also increased with successive testing as flaws were eliminated. The 0.2-percent yield and the tensile strength of cold-drawn and electropolished wires are presented as a function of inverse square root of wire diameter in figures 7 and 8, respectively. These least-squares plots have correlation coefficients of 0.91 and 0.97, respectively, indicating good fits to the square-root relation. It may be observed in figure 7 that these wires which were cold-drawn to 76.2 microns and electropolished to smaller sizes displayed a weak inverse dependence of proportional limit stress on diameter.

Examination of figure 8 shows there is no apparent dependence of tensile strength of the electropolished wires on diameter, as such, but that the strength observed is

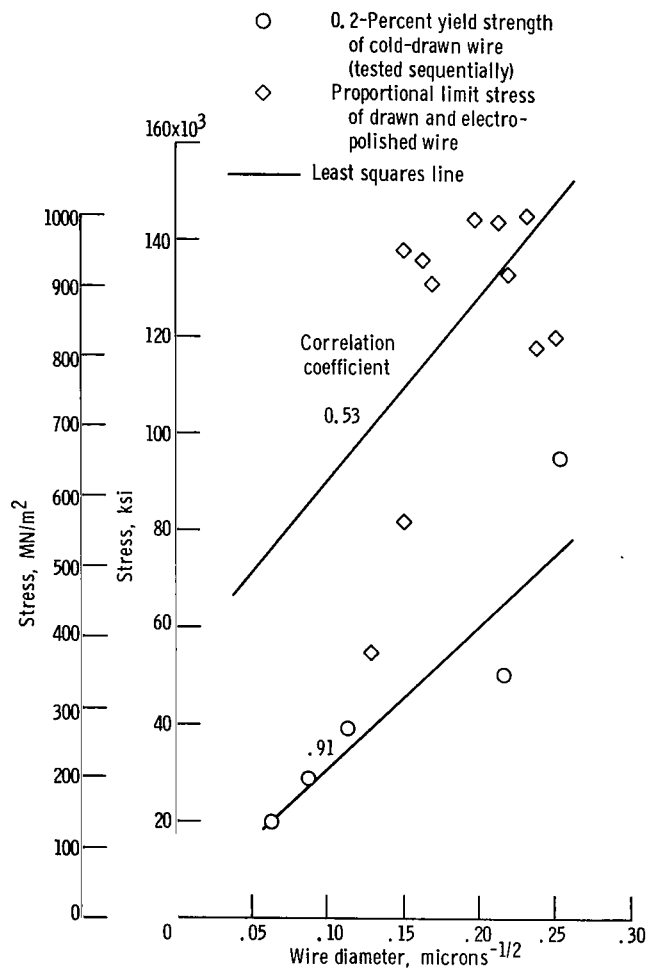


Figure 7. - Yield and proportional limit stress of nickel wires plotted against inverse square root of wire diameter.

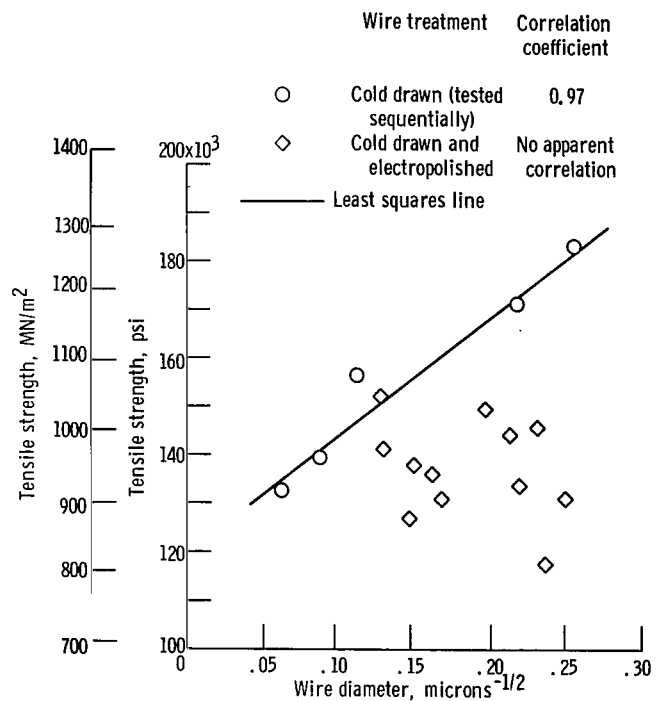


Figure 8. - Tensile strength of nickel wire plotted against inverse square root of wire diameter.



TABLE IV. - TENSILE DUCTILITY OF SMALL DIAMETER  
POLYCRYSTALLINE NICKEL WIRES

Cold drawn and electro-polished condition		Recrystallized and electropolished condition			
Wire diameter, micron	Total elongation, percent	Coarse-grain size		Fine-grain size	
		Wire diameter, micron	Total elongation, percent	Wire diameter, micron	Total elongation, percent
16.0	0.42	9.0	0.55	11.6	0.9
17.7	.50	12.9	2.1	21.1	2.7
18.7	.85	19.6	4.5	22.0	.21
20.6	.53	25.7	6.3	26.9	4.8
21.8	.60	29.7	8.2	30.6	5.9
25.2	.75	34.0	8.7	32.0	8.0
35.4	.60	69.7	16.1	32.3	11.6
37.3	.70			42.1	7.2
43.3	.55			42.9	11.0
44.8	.75				
60.4	1.40				

somewhat lower than the strength of the 76.2-micron-diameter wires in the as-cold-drawn condition. Total elongation at fracture of electropolished, cold-drawn wire was low and essentially size independent (table IV).

## DISCUSSION

The dependence of 0.2-percent yield strength of the cold-drawn wires on the inverse square root of wire diameter suggests that flow stress variations are affected by barrier spacings which, as expected, decrease with increasing amounts of strain during wire drawing. The strength of a dislocation barrier depends on the nature of the obstacle (ref. 19), while the stress concentration acting on the head of the dislocation pileup at the barrier is directly proportional to the number of dislocations in the procession and to the procession length. The important result is that the yield and tensile strengths of a material vary inversely with the square root of pileup length. Alternatively stated, it is the spacing between barriers that becomes important in determining the proportional limit or flow stresses.

It was recently shown, for the case in which the barriers were fragmented carbides in the cell walls, that the measured spacing was as small as 85 Å (or  $\times 10^{-10}$  m, ref. 17). This was consistent with the theoretical calculation of Armstrong, et al., who showed

that the Hall-Petch relation can be extended in a modified way to grain sizes (barrier spacings) smaller than  $50 \text{ \AA}$  (ref. 20). The applicability of this modified pileup theory for barrier dimensions, which are smaller than the diameters of the nickel wires of this study ( $15 \mu\text{m}$ ), was also shown for iron, which had grain sizes ranging from 1.6 to 400 microns (ref. 21).

Usually these analyses are applied to specimens that have cross sections consisting of 15 to 20 grain or cell diameters. In the present work this is true for the cold-drawn wires that were reduced in diameter by drawing. However, the recrystallized wires were reduced in diameter by electropolishing and do not satisfy these conditions. Thus, an analysis that considers the interaction of grain size and specimen diameter was required.

## Recrystallized Wires

In the RESULTS section it was reported (fig. 5) that both the coarse- and fine-grained electropolished material had proportional limit stresses that varied with wire diameter to a power of  $-1.22$  or  $-1.58$ , respectively. The dependence of proportional limit stress on wire diameter of the fine-grained material was greater than that of the coarse-grained, which suggests an interaction between wire diameter and grain size. Although only two grain sizes were available, it was considered worthwhile to evaluate the proportional limit stress against grain size in conformity to the Hall-Petch type of plot (refs. 22 and 23). This is presented in figure 9 for a number of different diameter wires. The data used for the wires with diameter less than 0.003 inch ( $76.2 \mu\text{m}$ ) were obtained from a solution of the least-squares equations (eqs. (3) and (4); see table II and fig. 5) for the electropolished material and from the averages of a number of individual tests at the conditions noted for the larger plain annealed wires. The curves when extrapolated to infinite grain size converge to a common point. The value of this apparent friction stress  $J_0$  agrees with the published values for single and polycrystalline nickel specimen (refs. 24 to 26). Additionally, the value of  $K_y$ , the unpinning stress, obtained from the slope of the Hall-Petch plot for the 254-micron-diameter wire ( $0.2 \text{ kg/mm}^{3/2}$ ), is consistent with those found for bulk nickel, copper, aluminum, and silver (refs. 25 and 27). Taking the apparent slopes of the lines for wires of different diameters and plotting them as an inverse function of wire diameter (fig. 10) we obtain a linear relation. If, as is observed, the principal contribution to the proportional limit arises from the effect of diameter on the unpinning term, then it is possible to evaluate the contribution of wire diameter to the proportional limit stress of nickel. This contribution is assumed to take

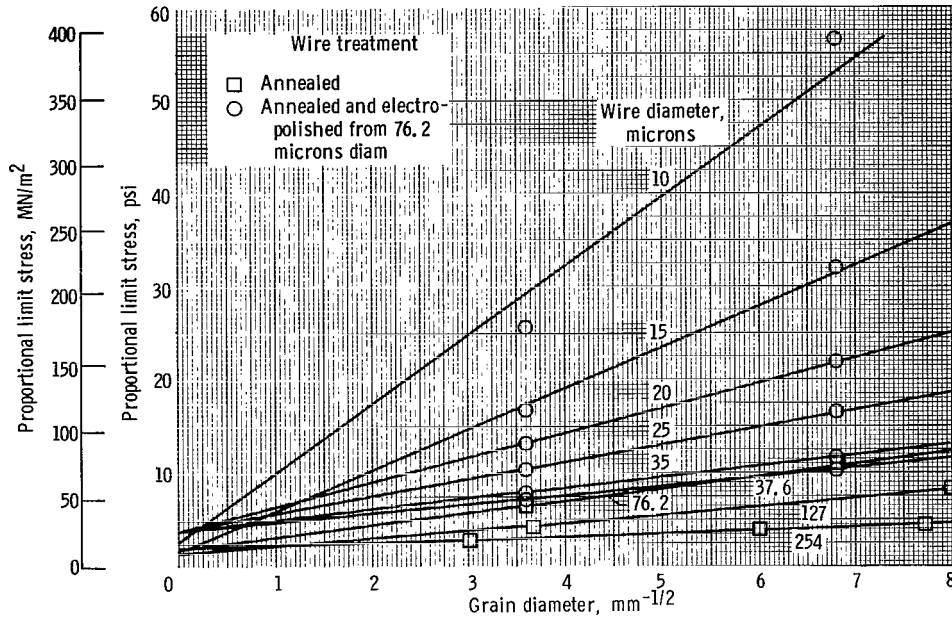


Figure 9. - Proportional limit stress against grain diameter for various diameter nickel wires.

the form of substituting a diameter dependent term into the Hall-Petch equation. This may be written as

$$J_{PL} = J_0 + K_y(D^{-1})L^{-1/2} \quad (5)$$

where  $J_{PL}$  is the proportional limit stress,  $J_0$  is the friction stress,  $K_y$  is the slope of the Hall-Petch plot,  $D$  is the wire diameter, and  $L$  is the grain size. From figure 10 we obtain

$$K_y = K_y(\text{bulk nickel}) + B/D \quad (6)$$

where

$$K_y^{10} \left( \begin{array}{c} \text{Bulk nickel} \\ \text{or} \\ 10\text{-mil (254-}\mu\text{m) wire} \end{array} \right) = 0.2 \frac{\text{kg}}{\text{mm}^{3/2}}$$

and

$$B = \text{Constant}$$

Therefore,

$$J_{PL} = J_0 + \left( K_y^{10} + \frac{B}{D} \right) L^{-1/2} \quad (7)$$

$$J_{PL} = J_0 + K_y^{10} L^{-1/2} + BD^{-1} L^{-1/2} \quad (8)$$

At constant diameter, equation (8) reduces to the strength-size dependence observed in figure 9. At constant grain size, equation (8) reduces to the strength-size dependence observed in figure 5. When diameter increases to a large value, such as 254 microns, the grain-size wire diameter-interaction term vanishes and equation (8) becomes the regular Hall-Petch relation. Finally, as the grain size approaches the wire diameter and the crystal approaches a single crystal, the proportional limit stress is modified by a contribution from diameter alone.

A comparison of the measured proportional limit stresses and the stresses obtained by calculation for the recrystallized and electropolished wires of different diameters is presented in figure 11. The calculated stresses were obtained by substituting into equation (8)  $J_0 = 3000$  psi (20.65 MN/m<sup>2</sup>),  $K_y$  for the appropriate diameter wire obtained from figure 10, and the pertinent grain sizes and diameters. Figure 11 shows that equation (8) adequately expresses the proportional limit stresses of small diameter, fine-grained recrystallized, and electropolished wires.

In summary, the proportional limit stress of recrystallized and electropolished fine-grain, small-diameter nickel wires may be considered to consist of three terms: a friction stress  $J_0$ , a grain size dependent term  $K_y L^{-1/2}$ , and a wire-diameter - grain-size interaction term  $BD^{-1} L^{-1/2}$ . In the recrystallized wires, where the diameter decreases

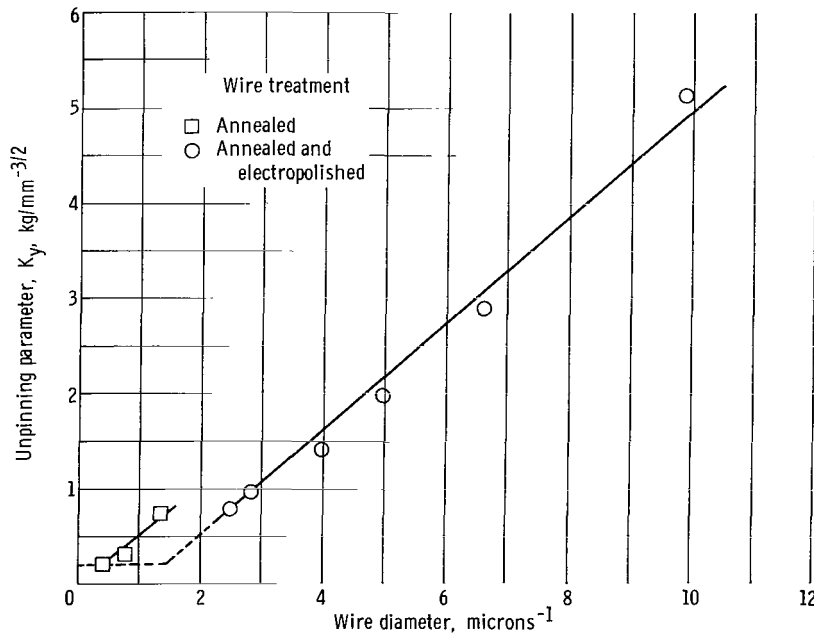


Figure 10. - Effect of wire diameter on unpinning parameter (slope of Hall-Petch plot).

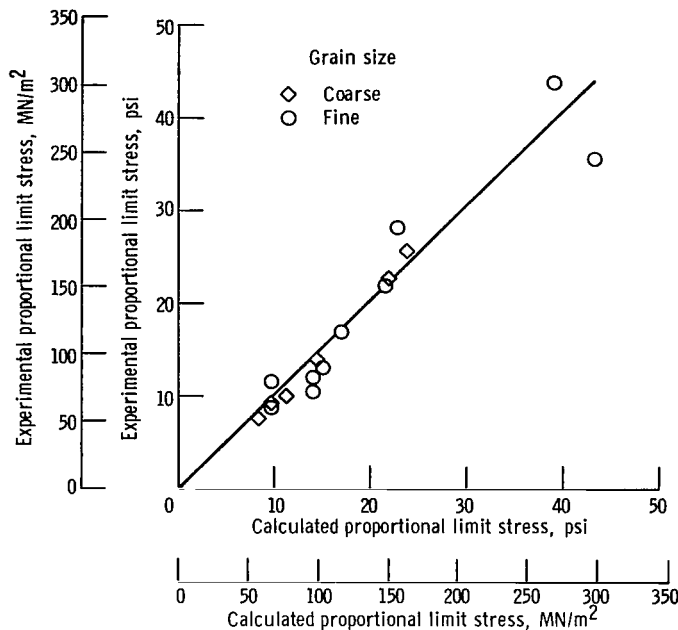


Figure 11. - Comparison of calculated and experimental proportional limit stresses.

due to electropolishing resulting in an increase in proportional limit stress, it is probable that the controlling dimension is the grain size until the wire diameter decreases to the same order of size or smaller than the grain size.

The dependence of  $K_y$  on the inverse of wire diameter suggests that the strength-wire size interaction may be explained in terms of surface effects such as contamination or high dislocation content debris layers (ref. 28) which impede the activation of dislocation sources. This is supported by figure 10 which shows that the unpinning stress for the recrystallized wires displays a higher value at a given diameter than the electropolished wires, which indicates that electropolishing was effective in lowering the stress required for source activation.

Initially, in larger diameter wires that have the same properties as bulk nickel, the grain size itself controls the deformation. However, when dimensions of the wires decrease and approach those of the starting grain size, the effects of surface on source activation becomes the controlling factor.

## Cold-Drawn Wires

In this study the use of a sequential testing technique established that the wires displayed a dependence of 0.2-percent (see figs. 7 and 8) yield and tensile strength on the

inverse square root of diameter. Thus, in the sequentially cold-drawn wires, the drawing deformation established a structure in which tensile strength depends primarily on a barrier that is sensitive to prior cold work rather than diameter. Although cell sizes were not determined, the strength-wire diameter interaction observed has been explained as being related to the substructure formed during drawing (ref. 17). For example, a linear relation between yield stress and inverse square root of substructure has been shown to hold for Ferrovac E iron (ref. 17). In this case the cells established during drawing were elongated so that the dimensions of the cells perpendicular to the wire axis was reduced in size. Continued deformation in the body centered cubic (BCC) metals continued to reduce the cell size.

In the case of face centered cubic (FCC) metals continued room-temperature deformation usually causes a decrease in cell size, which reaches a limiting dimension of approximately 0.5 micron (ref. 29). Hardness also reached a limiting value for nickel wires drawn to true strains of 3.5 (ref. 30). It is also consistent with the findings of Embury, et al., who showed that a cell structure formed in copper but that the cell size was not markedly reduced on further deformation (ref. 31). These authors suggested that the high energy associated with increasing dislocation contents led to dynamic recovery in the case of FCC metals and considered that in the case of BCC metals the cell walls are stabilized by impurities. However, for nickel, Nolder (ref. 32) found, in assessing the effects of explosive shock wave loading on the form of the stress-strain curve, that his stable cell sizes ranged down to 0.1 micron. In the present study the large drawing strains and the sequential testing may have aided in stabilizing the barriers against dynamic recovery.

## CONCLUSIONS

1. The proportional limit stress of recrystallized, small-diameter nickel wires may be expressed in terms of modified Hall-Petch equation, in which  $K_y$  is expressed as an inverse function of wire diameter, and the zero intercept  $J_0$  appears to be diameter independent

$$J_{PL} = J_0 + K_y^{10} L^{-1/2} + BD^{-1} L^{-1/2}$$

where  $J_{PL}$  is the proportional limit stress,  $J_0$  is the frictional stress or zero intercept of Hall-Petch plot,  $K_y$  is the slope of the Hall-Petch plot,  $L$  is the grain size,  $B$  is a constant, and  $D$  is the wire diameter.

2. The proportional limit stresses of recrystallized and electropolished nickel wires are linearly related to the inverse of wire diameter and may be represented by

$$J_{PL} = 4000 + 0.226 D^{-1.579}$$

and

$$J_{PL} = 3000 + 1.624 D^{-1.22}$$

for fine- and coarse-grained wires, respectively.

3. The tensile strength of recrystallized and electropolished wires appeared to be independent of diameter.

4. The 0.2-percent yield and tensile strengths of cold-drawn nickel wires are linearly related to the reciprocal square root of wire diameter.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, July 12, 1968,  
129-03-09-01-22.

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